



Ciba Geigy
received
02 Aug 91 MH
TRC/T6
9103021 IA

July 24, 1991

Dennis Gagne
Regional Sample Control Custodian
U.S. Environmental Protection Agency
90 Canal Street
Boston, MA 02114

REC'D
8-5-91
F.B.

Re: 68-W9-0003
Work Assignment R01005
Case 16142, SDG MAT341
Skinner and Sherman
Ciba-Geigy
Metals: 6/Soil, 1/Aqueous
Cyanide: 6/Soil, 1/Aqueous

Dear Mr. Gagne:

The following is a Data Validation Report for CLP case 16142, which was generated by QuantaLex Inc., Alliance's Data Validation Sub-Contractor for this work assignment. The inorganic analytical data for this case contained low level water and soil samples which were collected by Alliance Technologies Corporation at the Ciba-Geigy Site and analyzed by Skinner and Sherman.

If you have any questions, please feel free to contact me at (508) 970-5600 X 4201.

Sincerely,

Cynthia S. Fortin
Data Validation Coordinator

encl.

cc: Deborah Szaro/Moira Lataille, Region I TPO



SEMS DocID

666773

May 30, 1991

Ms. Cynthia Fortin
Data Validation Coordinator
Alliance Technologies Corp.
Boott Mills South, Foot of John Street
Lowell, MA 01852

Re: Case 16142, SDG MAT341
Skinner & Sherman
Metals: 6/Soil, 1/Aqueous Sample
Cyanide: 6/Soil, 1/Aqueous Sample

Dear Ms. Stallings:

A validation was performed on the analytical data from six low level soil and one low aqueous samples which were collected by Alliance Technologies Corp. and submitted to Skinner & Sherman for Inorganic analysis. The data were evaluated based on the following parameters:

- * ■ Data completeness
- * ■ Holding times
- Calibration verification
- Laboratory and field blank analyses
- * ■ ICP interference check sample results
- Matrix spike recoveries
- * ■ Laboratory and field duplicates
- * ■ Laboratory control sample results
- Furnace atomic absorption results
- * ■ Serial dilution results
- * ■ Detection limit results
- * ■ Sample results

* - All criteria were met for this parameter

Table 1 summarizes the validation recommendation which were based on the following information.

Calibration Verification

The Selenium initial calibration correlation coefficient was less than 0.995. The positive value for MAT343 is estimated (J).

Blanks

<u>Element</u>	<u>Maximum Conc./Units</u>	<u>Action Level/ Units</u>
Cu	24.3 ug/L	121.5 ug/L
Ni	26.0 ug/L	130 ug/L
Na	1245 ug/L	1225 ug/L
As	1.3 ug/L	6.5 ug/L

Value > IDL, < CRDL, and < Action Level = Report value estimated (UJ).

Value > IDL, > CRDL, and < Action Level = Report value U.

Value > IDL and > Action Level = Report value unqualified.

Matrix Spike

<u>Analyte</u>	<u>SSR</u>	<u>SR</u>	<u>S</u>	<u>%R</u>
Hg	0.7903 ug/L	0.1174 U ug/L	0.56 ug/L	141

Positive results are estimated (J) and non-detects are acceptable (A) when spike recovery is greater than 125%.

MSA Results

<u>ITR #</u>	<u>Analyte</u>	<u>Correlation Coefficient #1</u>	<u>Correlation Coefficient #2</u>
MAT343	Se	0.9408	0.9916

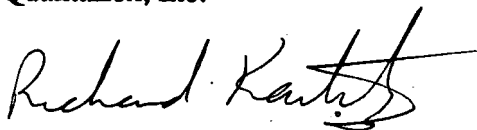
Estimate (J) positive results.

General Comments

The incorrect percent solids was used to calculate the duplicate results.

The incorrect percent solids was used for calculating the final result for MAT343. All results reported in the tables were calculated using the correct value of 51.4%.

Very truly yours,
QuantaLex, Inc.



Richard Kantrowitz
Associate Consultant

cc: Keith Wegner, President

**SKINNER & SHERMAN
CASE 16142**

TABLE I - RECOMMENDATIONS SUMMARY

Aluminum	A	Magnesium	A
Antimony	A	Manganese	A
Arsenic	J2	Mercury	J1
Barium	A	Nickel	J2,A1
Beryllium	A	Potassium	A
Cadmium	A	Selenium	J3
Calcium	A	Silver	A
Cobalt	A	Sodium	J2
Chromium	A	Thallium	A
Copper	A1	Vanadium	A
Iron	A	Zinc	A
Lead	A	Cyanide	A

- A - Accept all data.
- A1 - Accept data, raise the sample detection limit(s) due to blank contamination.
- J1 - Estimate (J) positive values due to poor pre-digestion matrix spike recovery.
- J2 - Estimate (UJ) positive values due to blank contamination and values < CRDL.
- J3 - Estimate (J) positive value due to the initial calibration and the MSA correlation coefficient < 0.995.

INORGANIC REGIONAL DATA ASSESSMENT

Region I

CASE NO. 16142
 LABORATORY Skinner & Sherman
 SDG # MAT341
 SOW # 7/88
 DPO: ACTION _____ FYI _____

SITE Ciba-Geigy
 NO. OF SAMPLES/
 MATRIX 6/Water, 1/Soil
 REVIEWER (IF NOT ESD) QuantaLex, Inc.
 REVIEWER'S NAME Richard Kantrowitz
 COMPLETION DATE May 30, 1991

Data Assessment Summary

	ICP	AA	Hg	Cyanide
1. Holding Times	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>
2. Calibrations	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>
3. Blanks	<u>X</u>	<u>O</u>	<u>O</u>	<u>O</u>
4. ICS	<u>O</u>	<u>N/A</u>	<u>N/A</u>	<u>N/A</u>
5. LCS	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>
6. Duplicate Analysis	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>
7. Matrix Spike	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>
8. MSA	<u>N/A</u>	<u>M</u>	<u>N/A</u>	<u>N/A</u>
9. Serial Dilution	<u>O</u>	<u>N/A</u>	<u>N/A</u>	<u>N/A</u>
10. Sample Verification	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>
11. Other QC	<u>X</u>	<u>X</u>	<u>X</u>	<u>X</u>
12. Overall Assessment	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>

O = Data had no problems/or qualified due to minor problems.

M = Data qualified due to major problems.

Z = Data unacceptable.

X = Problems, but do not affect data.

ACTION ITEMS: MSA correlation coefficients < 0.995.

AREAS OF CONCERN: 1) Contaminates in the rinsate blank.

2) Incorrect percent solids used for MAT343.

NOTABLE PERFORMANCE: _____

REGION I REVIEW OF INORGANIC
CONTRACT LABORATORY DATA PACKAGE

The hardcopied Skinner & Sherman data package received at Region I has been reviewed and the quality assurance and performance data summarized. The data reviewed included:

Case No. 16142 SAS No. _____ Sampling Date(s): 03/28/91
SDG No. MAT341 Matrix Soil/Water Shipping Date(s): 03/28/91
No. of Samples 7 Date Rec'd by Lab: 03/29/91

Traffic Report Nos.: MAT341, MAT342, MAT343, MAT344, MAT345, MAT346,
MAT347

Trip Blank No.: _____

Equipment Blank No.: MAT341

Field Dup. Nos.: MAT343, MAT344

SOW No. 7/88 requires that specific analytical work be done and that associated reports be provided by the laboratory to the Regions, EMSL-LV, and SMO. The general criteria used to determine the performance were based on an examination of:

- | | |
|----------------------------------|-------------------------------|
| - Data Completeness | - Field Duplicates |
| - Holding Times | - Lab Control Sample Results |
| - Calibrations | - Furnace AA Results |
| - Blanks | - ICP Serial Dilution Results |
| - ICP Interference Check Results | - Detection Limit Results |
| - Matrix Spike Recoveries | - Sample Quantitation |
| - Laboratory Duplicates | |

Overall comments: Data acceptable with qualifications.

Definitions of Qualifiers:

- A - Acceptable data.
J - Approximate data due to quality control criteria.
R - Reject data due to quality control criteria.
U - Compound not detected.

Reviewer: Richard K. [Signature]

Date: 5/30/91

REGION I

Data Review Worksheets

I. DATA COMPLETENESS

MISSING INFORMATION

DATE LAB CONTACTED

DATE RECEIVED

None

II. HOLDING TIMES

Complete table for all samples and circle the fractions which are not within criteria.

SAMPLE ID	DATE SAMPLED	Hg DATE ANAL	CYANIDE DATE ANAL	OTHERS DATE ANAL	pH	ACTION
MAT341	03/28/91	04/17/91	04/09/91	04/18/91		None
MAT342	03/28/91	04/17/91	04/09/91	04/18/91		None
MAT343	03/28/91	04/17/91	04/09/91	04/18/91		None
MAT344	03/28/91	04/17/91	04/09/91	04/18/91		None
MAT345	03/28/91	04/17/91	04/09/91	04/18/91		None
MAT346	03/28/91	04/17/91	04/09/91	04/18/91		None
MAT347	03/28/91	04/17/91	04/09/91	04/18/91		None

METALS - 180 days from sample collection
MERCURY - 28 days from sample collection
CYANIDE - 14 days from sample collection

ACTION:

1. If holding times are exceeded, all positive results are estimated (J) and non-detects are estimated (UJ).
2. If holding times are grossly exceeded, the reviewer may determine that non-detects are unusable (R).

III A. INSTRUMENT CALIBRATION (Section 1)

1. Recovery Criteria

List the analytes which did not meet the percent recovery (%R) criteria for Initial or Continuing Calibration.

<u>DATE</u>	<u>ICV/CCV#</u>	<u>ANALYTE</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
<u>None</u>					

ACTIONS:

If any analyte does not meet the %R criteria, follow the actions stated below:

For Positive Results:

	<u>Accept</u>	<u>Estimate (J)</u>	<u>Reject (R)</u>
Metals	90-110%R	75-89%R, 111-125%R	<75%R, >125%R
Mercury	80-120%R	65-79%R, 121-135%R	<65%R, >135%R
Cyanide	85-115%R	70-84%R, 116-130%R	<70%R, >130%R

For Non-detected Results:

	<u>Accept</u>	<u>Estimate (UJ)</u>	<u>Reject (R)</u>
Metals	90-125%R	75-89%R	<75%R, >125%R
Mercury	80-135%R	65-79%R	<65%R, >135%R
Cyanide	85-130%R	70-84%R	<70%R, >130%R

III B. INSTRUMENT CALIBRATION (Section 2)

2. Analytical Sequence

- | | | |
|----|---|-----|
| A. | Did the laboratory use the proper number of standards for calibration as described in the SOW? | Yes |
| B. | Were calibrations performed at the beginning of each analysis? | Yes |
| C. | Were calibration standards analyzed at the beginning of sample analysis and at a minimum frequency of ten percent or every two hours during analysis, whichever is more frequent? | Yes |
| D. | Were the correlation coefficients for the calibration curves for AA, Hg, and CN ≥ 0.995 ? | No |
| E. | Was a standard at 2xCRDL analyzed for all ICP analyses? | Yes |

If no,
the data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

The Zn CRDL check sample recovery was 131.7%. No samples were affected.

Se (run 2) initial calibration correlation coefficient was 0.9942.

REGION I
Data Review Worksheets

IV A. BLANK ANALYSIS RESULTS (Sections 1-3)

List the blank contamination in Sections 1 and 2 below. A separate worksheet should be used for soil and water blanks.

1. Laboratory Blanks

Matrix: Soil

<u>DATE</u>	<u>ICB/CCB#</u>	<u>PREP BL</u>	<u>ANALYTE</u>	<u>CONC./UNITS</u>
04/17/91	CCB2		Al	-24.2 ug/L
04/17/91	ICB		Ca	-24.9 ug/L
04/17/91	CCB2		Cu	24.3 ug/L
04/17/91	ICB		Fe	-11.8 ug/L
04/17/91	CCB3		Mn	2.0 ug/L
04/17/91		PBS	Ni	26.0 ug/L
04/17/91	ICB		Na	-32.6 ug/L
04/17/91		PBS	Zn	17.0 ug/L
04/17/91	CCB4		Pb	1.5 ug/L

2. Equipment/Trip Blanks

<u>DATE</u>	<u>EQUIP BL#</u>	<u>ANALYTE</u>	<u>CONC./UNITS</u>
04/18/91	MAT341	As	1.3 ug/L
04/17/91	MAT341	Ca	34.0 ug/L
04/17/91	MAT341	Cr	5.7 ug/L
04/17/91	MAT341	Cu	7.1 ug/L
04/17/91	MAT341	Fe	34.7 ug/L
04/18/91	MAT341	Pb	2.0 ug/L
04/17/91	MAT341	Mn	1.1 ug/L
04/17/91	MAT341	Na	245 ug/L
04/17/91	MAT341	Zn	24.6 ug/L

3. Frequency Requirements

- A. Was a preparation blank analyzed for each matrix, for every 20 samples, and for each digestion batch? Yes
- B. Was a calibration blank run every 10 samples or every 2 hours, whichever is more frequent? Yes

If no,

the data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

IV B. BLANK ANALYSIS RESULTS (Section 4)

4. Blank Actions

The Action Levels for any analyte is equal to five times the highest concentration of that element's contamination in any blank. The action level for samples which have been concentrated or diluted should be multiplied by the concentration/dilution factor. No positive sample result should be reported unless the concentration of the analyte in the sample exceeds the Action Level (AL). Specific actions are as follows:

1. When the concentration is greater than the IDL, but less than the Action Level, report the sample concentration detected with a U.
2. When the sample concentration is greater than the Action Level, report the sample concentration unqualified.

MATRIX: Soil

<u>ELEMENT</u>	<u>MAX. CONC./ UNITS</u>	<u>AL/ UNITS</u>
<u>Cu</u>	<u>24.3 ug/L</u>	<u>121.5 ug/L</u>
<u>Ni</u>	<u>26.0 ug/L</u>	<u>130 ug/L</u>
<u>Na</u>	<u>245 ug/L</u>	<u>1225 ug/L</u>
<u>Zn</u>	<u>24.6 ug/L</u>	<u>123 ug/L</u>
<u>As</u>	<u>1.3 ug/L</u>	<u>6.5 ug/L</u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>

MATRIX:

<u>ELEMENT</u>	<u>MAX. CONC./ UNITS</u>	<u>AL/ UNITS</u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
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<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>

NOTE: Blanks analyzed during a soil case must be converted to mg/kg in order to compare them with the sample results.

$$\text{Conc. in ug/L} \times \frac{\text{Volume diluted to (200 ml)}}{\text{Weight digested (1 gram)}} \times \frac{1\text{L}}{1000\text{ml}} \times \frac{1000\text{ gm}}{1\text{ kg}} \times \frac{1\text{ mg}}{1000\text{ug}} = \text{mg/kg}$$

Multiplying this result by 5 to arrive at the action level gives a final result in mg/kg which can then be compared to sample results.

V A. ICP INTEFERENCE CHECK SAMPLE (Sections 1 and 2)

1. Recovery Criteria

List any elements in the ICS AB solution which did not meet the criteria for %R.

<u>DATE</u>	<u>ELEMENT</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
<u>None</u>				

ACTIONS:

If an element does not meet the %R criteria, follow the actions stated below:

	<u>Percent Recovery</u>		
	<u><50%</u>	<u>50%-79%</u>	<u>>120%</u>
Positive sample results	R	J	J
Non-detected results	R	UJ	A

2. Frequency Requirements

Were interference QC samples run at the beginning and end of each sample analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent?

Yes

If no,

the data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.
